

Major Digestion Method

1. Introduction

The Major Digestion Method is a dissolution process that is conducted in order to chemically dissolve solid sample material into a solution containing major elements in preparation for analysis on the ICP-OES. This process also eliminates or sequesters any remaining organics in the samples.

This method is similar to the dissolution described in the ASTM Method D 6349-07 section 10.3.2

2. Interfaces with Other Methods

This method relies on:

EGL Method 29, Calibration of Laboratory Scales and Analytical Balances and
EGL Method 25, Method for Sample Login, Control, and Disposition.

This method prepares digested samples for:

EGL Method 16, The determination of Major/Minor Elements in Geological Samples by Inductively-Coupled Plasma Optical Emission Spectrometry.

3. Materials and Equipment

Hotblock¹ capable of heating up to 90°C, 50 ml polypropylene digestion tubes, deionized water (DI H₂O), nitric acid (HNO₃), hydrochloric acid (HCl), hydrofluoric acid (HF) 30% reagent grade or better hydrogen peroxide (H₂O₂), 1.5% boric acid (H₃BO₃) – in a 2000ml volumetric flask, add 30g of H₃BO₃ and dilute to volume with DI water, disposable chemical resistant gloves, eye protection, and lab coat.

4. Procedure

Weigh out approximately 0.1 grams of the sample material (laboratory prepared ash 750°C or LOI 750°C depending on original sample type) into a 50 ml digestion tube. Record the mass of the sample for future reference and for use in the ICP-OES method.

Add 1 ml of concentrated HNO₃ to the samples followed by 2.5 ml of a premixed 70/30 HCl/HF solution. Next add 1 ml of H₂O₂ and allow to rest until the reaction is complete. Mix the digestion tubes well and cap them securely. Place the tubes in a hotblock preheated to approximately 90°C and maintain at this temperature for approximately 2.5 hours. After the allotted time, remove the tubes and cool for around 15 minutes.

¹ Any use of trade names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

Once the samples are sufficiently cooled, add 45.5 ml of the prepared H_3BO_3 (for a final volume of 50 ml) and recap the digestion tubes. Return the samples to the hotblock which is still set to approximately 90°C for about 1 hour. Remove the digestion tubes from the hotblock and allow them to sit overnight before analyzing on the ICP-OES.

5. Calibration and Quality Control Samples

Check the temperature of the hotblock periodically with a thermometer. Adjust the temperature control on the front of the hotblock if necessary. Digest current, approved lab standards, blanks, and duplicate samples in the same manner as described above.

6. Limits, Precautions, and Interferences

As a precaution, place the hotblock under a hood to help dissipate heat and fumes. Place all acids under a hood and perform all the dispensing of the acids under a hood. During the entire process, all safety gear should be worn.

7. Acceptance of Data

This procedure does not produce any data and therefore acceptance of data does not apply.

8. Data Handling and Transfer

The sample masses are transferred from the balance to an Excel™ template electronically which is saved on the shared network drive.

9. References

American Society for Testing and Materials International [ASTM], 2007, Annual book of ASTM standards, section five, petroleum products, lubricants, and fossil fuels, Gaseous fuels; coal and coke: West Conshohocken, Pennsylvania, American Society for Testing and Materials International, v. 05.06, p. 602.

10. Attachments

None

11. History of Changes

Revision 0: initial issue